KOKAI PATENT APPLICATION NO. HEI 7-286166

ALUMINA-ZIRCONIA QUALITY SINTERED ABRASIVE GRAINS AND MANUFACTURING METHOD THEREOF

[Translated from Japanese]

[Translation No. LPX20357]

Low ZrO2 content

Translation Requested by: Paul Davies 3M

Translation Provided by: Yoko and Bob Jasper
Japanese Language Services
16 Oakridge Drive
White Bear Lake, MN 55110

(651) 426-3017 Fax (651) 426-8483
e-mail: bjasper@attbi.com

JAPANESE PATENT OFFICE (JP)

PATENT JOURNAL (A)

KOKAI PATENT APPLICATION NO. HEI 7-286166

Technical Indication Section

Int. Cl.^{6}:	C 09 K 3/14 B 24 D 3/00 C 04 B 35/10
Identification Code:	550 D H 320 A 3
Sequence Nos. for Office Use:	FI
Filing No.:	Hei 6-80638
Filing Date:	April 19, 1994
Publication Date:	October 31, 1995
No. of Claims:	3 OL (Total of 5 pages in the [Japanese] document)
Examination Request:	Not filed

ALUMINA-ZIRCONIA QUALITY SINTERED ABRASIVE GRAINS AND MANUFACTURING METHOD THEREOF

[*Arumina-girukonia shitsu shohketsu toryuh oyobi sono seizoh houhoh*]

Inventor(s): Hisashi Morimoto
c/o Showa Denko Co., Ltd.
Shiojiri Plant
1 Soga, Oaza
Shiojiri-shi, Nagano-ken

Yukiteru Kaneko
c/o Showa Denko Co., Ltd.
Shiojiri Plant
1 Soga, Oaza
Shiojiri-shi, Nagano-ken

Applicant(s): 000002004
Showa Denko Co., Ltd.
1-13-9 Daimon
Shiba, Minato-ku
Tokyo

Agent(s):

[There are no amendments to this patent.]

[*Translator's note: Names of products and companies are spelled phonetically in this translation.*]

[*Translator's note: The format of the source document supplied by the requester has been changed from the original Japanese Patent Application submitted by the applicant to accommodate machine translation.*]

[Title of the invention]

Alumina-zirconia quality sintered abrasive grains and manufacturing method thereof

[Abstract]

[Purpose] The purpose of the present invention is to produce alumina-zirconia quality sintered abrasive grains capable of maintaining high hardness and having high toughness and

high abrasion resistance and high grinding ratio.

[Constitution] Alumina-zirconia quality sintered abrasive grains containing a small amount of SiO_2, and having a friability value that indicates a toughness value of 15 or less and micro-Vicker's hardness of at least 1800 kg/mm^2.

[Claims of the invention]

[Claim 1] Alumina-zirconia quality sintered abrasive grains essentially made of an alumina-zirconia quality composition containing

(1) 3 to 15 wt% of ZrO_2,

(2) 0.05 to 3.0 wt% of SiO_2,

(3) 0.1 to 3.0 wt% of MgO, CoO, or NiO as the indication conversion, and having a friability value that indicates a toughness value of 15 or less and micro-Vicker's hardness (Hv500) under load of 500 g of at least 1800 kg/mm^2.

[Claim 2] A manufacturing method of alumina-zirconia quality sintered abrasive grains characterized by the fact that 3 to 15 wt% of zirconia, 0.05 to 3.0 wt% of a clay as SiO_2 in the sintered abrasive grain, and 0.1 to 3 wt% of either magnesium oxide, cobalt oxide, or nickel oxide is added for an alumina fine powder with a purity of at least 98%, mixing is performed and molding is done, and baking is further provided at a temperature in the range of 1550 to 1750°C.

[Claim 3] The manufacturing method of alumina-zirconia quality sintered abrasive grains described in claim 2 in which the clay is at least one type selected from the group consisting of bentonite, kibushi clay, frog eye clay, stoneware clay and sericite clay.

[Detailed description of the invention]

[0001]

[Field of industrial application] The present invention pertains to alumina-zirconia quality sintered abrasive grains with high toughness suitable for heavy grinding.

[0002]

[Prior art] In the past, for grinding powders used for heavy grinding, abrasive grains produced by sintering a fine powder of an alumina such as bauxite (Japanese Kokoku [Examined] Patent Application No. Sho 39-4398, Japanese Kokoku [Examined] Patent Application No. Sho 39-27612, Japanese Kokoku [Examined] Patent Application No. Sho 39-27613, and Japanese Kokoku [Examined] Patent Application No. Sho 39-27614), a molten alumina-zirconia abrasive grains (Japanese Kokoku [Examined] Patent Application No. Sho 39-16592), a molten alumina-zirconia abrasive grains containing SiO_2, TiO_2, MgO, CaO, etc. (Japanese Kokoku [Examined] Patent Application No. Sho 50-13989), an abrasive grains produced by adding a crystal growth inhibitor such as magnesium oxide to a high-purity alumina fine powder (Japanese Kokai [Unexamined] Patent Application No. Sho 52-14993), and an abrasive grains produced by adding a crystal growth inhibitor such as magnesium oxide and a clay for reducing internal cracks in the abrasive grains as a silicon dioxide to a high-purity alumina fine powder (Japanese Kokai [Unexamined] Patent Application No. Hei 4-20586) are known. Furthermore, an alumina quality sintered abrasive grains containing zirconia, hafnia, nickel, cobalt, zinc magnesia, etc. based on sol-gel method is known as well.

[0003]

[Problems to be solved by the invention] In bauxite type sintered abrasive grains, in addition to approximately 85% of alumina && (Al_2O_3), impurities such as TiO_2, Fe_2O_3 and SiO_2 are included in bauxite (calcinated) used, and wear rate is high, but hardness is low. In fused alumina-zirconia abrasive grains quenched to solidify after melting, crushed, and granulated to produce a product, and production with uniform grit at a high yield is difficult. Furthermore, high-purity alumina fine powder used for heavy grinding exhibits high hardness and high mechanical strength, but toughness is inadequate, and grinding ratio per hour is high, but abrasion resistance is low; thus, a wide range of application is not possible. The purpose of the present invention is to eliminate the above-mentioned existing problems, and to retain a minimum hardness, to increase abrasion resistance through an increase in the toughness of the

abrasive grains and to increase the grinding ratio, and molding is performed to produce grains of the required size by means of extrusion, etc. and to prevent formation of unnecessary grain size.

[0004]

[Means to solve the problem] As a result of much research conducted by the present inventors in an effort to eliminate the above-mentioned existing problems, the present invention was accomplished. In other words, the present invention is alumina-zirconia quality sintered abrasive grains essentially made of an alumina-zirconia quality composition containing

(1) 3 to 15 wt% of ZrO_2,

(2) 0.05 to 3.0 wt% of SiO_2,

(3) 0.1 to 3.0 wt% of MgO, CoO, or NiO as the indication conversion, and having a friability value that indicates a toughness value of 15 or less and micro-Vicker's hardness (Hv500) under a load of 500 g of at least 1800 kg/mm^2, and to provide a method of manufacturing the alumina-zirconia quality sintered abrasive grains that is characterized by the fact that 3 to 15 wt% of zirconia, 0.05 to 3.0 wt% of a clay as SiO_2 in the sintered abrasive grain, and 0.1 to 3 wt% of at least one of magnesium oxide, cobalt oxide, or nickel oxide is added for an alumina fine powder with a purity of at least 98%, mixing is performed and molding is carried out, and baking is further done at a temperature in the range of 1550 to 1750°C, and furthermore, the manufacturing method of alumina-zirconia quality sintered abrasive grains described in claim 2 in which the clay is at least one type selected from the group consisting of bentonite, kibushi clay, frog eye clay, stoneware clay and sericite clay.

[0005] The purity of the alumina raw material fine powder used in the present invention is at least 98 wt%, preferably, at least 99 wt%, and granularity is preferably 10 μm or below with a mean particle diameter of d50, preferably, a fine powder of 2 μm or below. When the purity is 98 wt% or below, adjustment of other components is difficult and uniformity is difficult to achieve. On the other hand, when the granularity exceeds 10 μm, a sinter with high-density is less likely to be produced, and the crystal size of the sinter is increased and mechanical strength

is reduced. Furthermore, the zirconia used in the present invention is at least 98 wt%, and a fine powder with a granularity of 10 μm or below, preferably, 1 μm or below, as d50. The amount of the zirconia component added is in the range of 3 to 15 wt%, and when 3 wt% or below, the effect of increase in the abrasive grains is insufficient; on the other hand, when the amount exceeds 15 wt%, a significant reduction in hardness occurs. Furthermore, as a crystal inhibitor of abnormal grain, 0.1 to 3.0 wt% of at least one type selected among the group of magnesium oxide, cobalt oxide, or nickel oxide is added at the time of sintering. The effect achieved is insignificant when the amount included is 0.1 wt% or below; on the other hand, when the amount exceeds 3.0 wt%, hardness of the abrasive grains is reduced, and mechanical strength is reduced as well. It is desirable when the purity of the above-mentioned crystal inhibitor is at least 98 wt% and d50 granularity is 10 μm or below, preferably, 5 μm or below. In the present invention, in order to form a column-like abrasive grains, it is desirable when 0.2 to 3.0 wt% of an organic binder such as PVA and methyl cellulose (MC), or preferably an aqueous solution of these is used, but adequate shape retention cannot be achieved by itself; thus, addition of a clay is essential in the present invention so as to increase the plasticity, and softness of the mixture, and to reduce microcracks in the molded article.

[0006] Many different types of clays are available, and from the standpoint of an increase in plasticity of the kneaded material of the present invention, at least one type selected from the group consisting of bentonite, kibushi clay, frog eye clay, stoneware clay and sericite clay is desirable. And based on the test results described below, the effectiveness is in the order of bentonite > sericite clay > kibushi clay > frog eye clay > stoneware clay from the standpoint of plasticity and strength after drying and bentonite is especially desirable. In comparison to the case where a clay is not included, even addition of stoneware clay exhibits higher extrusion moldability, and from the standpoint of extrusion moldability alone, bentonite is especially desirable. The amount of the clay added is in the range of 0.05 to 3.0 wt% in terms of the SiO_2 in the sintered abrasive grains, and when less than 0.05 wt%, the increase in plasticity and

flexibility of the kneaded material is inadequate; on the other hand, when the amount of exceeds 3.0 wt%, formation of a vitreous material or mullite-like material occurs and wear rate of the abrasive grains is significantly reduced, and an increase in the crystal growth due to sintering leads to a decrease in strength.

[0007] In the present invention, mixing is performed for the above-mentioned raw materials, namely, alumina fine powder, zirconia, clay and an abnormal crystal inhibitor such as magnesium oxide at the above-mentioned range. In this case, it is desirable when an aqueous solution or nonaqueous solution of organic binder such as PVA is added as described above, thorough kneading is performed and extrusion is performed by an extruder, etc. and molding is performed to form a column, and the molding method is not limited to extrusion method. Furthermore, drying is performed at a temperature in the range of 100 to 150°C and sintering is performed. For the sintering temperature, a temperature in the range of 1550 to 1750°C is suitable, and when the temperature is 1550°C or below, it is not possible to produce a high-density sinter; on the other hand, when the temperature exceeds 1750°C, the crystal size becomes too large and toughness and mechanical strength are inadequate. For production of abrasive grains, crushing or granulation is performed after sintering, or crushing or granulation is performed after drying and sintering is performed to form a predetermined granularity.

[0008] Alumina-zirconia quality sintered abrasive grains produced by the above-mentioned manufacturing method of the present invention is described below. The abrasive grains of the present invention have a composition with the raw material mixing ratio described above, namely, essentially an alumina-zirconia quality composition, and further includes 3 to 15 wt% of ZrO_2, 0.05 to 3.0 wt% of SiO_2, and 0.1 to 3.0 wt% of either CoO or NiO. Abrasive grains having the above-mentioned composition are known, but production of an abrasive grains with high toughness and high strength is made possible for the first time based on the unique method of the present invention. In other words, it is possible to produce alumina-zirconia quality sintered abrasive grains having a friability value that indicates a toughness value of 15 or less and micro-

Vicker's hardness (Hv500) under a load of 500 g of at least 1800 kg/mm^2.

[0009] Furthermore, the measurement method of the above-mentioned friability value is described below. The aforementioned measurement method is the method specified in JIS R6128 (Test Method for Toughness of Artificial Abrasives), but an adjustment was made for the conditions as shown below.

(1) Approximately 300 g of sample abrasive grains is measured and screening is performed for 5 minutes by a standard sieve net of each granularity specified by the JIS using ro-tap tester. Screening is further performed for the entire amount of sample remaining on the third step sieve for 5 minutes and the sample remaining on the third step sieve is used as the sample.

(2) 100 g of test sample is measured accurately at plus-minus 0.1 g, poured into a liter ball mill with 114 mm diameter, and 2 kg of steel balls with 3/4 inch is further poured into the ball mill.

(3) Crushing is performed for the test sample for 15 minutes at a mill rotation rate of 95 rpm. Subsequently, removal of the contents in the ball mill is performed and crushed sample was recovered.

[0010] (4) Screening is performed for the crushed sample for 5 minutes by the above-mentioned standard sieve net using a ro-tap tester.

(5) The test sample remaining in each sieve is measured accurately. The total amount of the sample remaining on first to fourth sieves is deducted from 100 g of test sample and the value obtained is defined as the friability value. In other words, the negative mesh of the fourth step sieve becomes the friability value, and the smaller said value, the higher the toughness.

[0011]

[Working Examples] In the following, the present invention is further explained in detail with working examples and comparative examples.

Working example 1

For crushed powder of sintered alumina (purity 99.5%, specific surface area 5 m^2/g,) (d50=1.99 μm, d10=0.44 μm, d90=7.76 μm), 3 wt% of zirconia (product of Showa Denko Co., Ltd., RZ-N,

#6000F, d50=0.89 μm) and 0.3 wt% of bentonite (product of Kanto Bentonite and Minerals Co., (Ltd.), Tenryu) were added, and furthermore, 0.2 wt% of magnesium oxide (product of Kyowa Chemical Co., Ltd., Kyowa-Mag 30, purity 99 wt%, d50=4.51 μm) was added, and 0.4 wt% of PVA was further added as a binder; then, 26 wt% of water was added for the total weight and kneading was performed by a mix muller, and furthermore, extrusion was performed from a die having a pore diameter of 2.2 mm to form abrasive grains with a #12 granularity as specified by JIS R6001-1987, and drying was further performed for 1 hour at a temperature of 150°C. The dried product was cut to form a length of 2 to 6 mm, and baking was performed for 1 hour at 1700°C in a rotary kiln.

[0012] Properties of the abrasive grains produced as described above are shown in Table II below. In order to examine cracks inside the grain, the column was cut at the center in the length direction and the cross-section was observed by a microscope at a magnification of 10 times. The ratio of grains that include even a single crack was calculated as the crack formation ratio. Furthermore, the compressive fracture strength is a value obtained based on [fracture load]/[projected surface area of the abrasive grains], and furthermore, the crystal size was measured by SEM.

[0013] Comparative example 1

As shown in Table I, addition of bentonite was omitted and production of an abrasive grains with granularity of #12 was produced as in the case of the above-mentioned working example 1. Properties are shown in Table II. The amount of SiO_2 included in the abrasive grains is based on impurities in the alumina raw material.

[0014] Comparative example 2

As shown in Table I, addition of ZrO_2 was omitted and production of an abrasive grains with a granularity of #12 was produced as in the case of the above-mentioned working example 1. Properties are shown in Table II.

[0015] As shown in Table II, the value of friability is very low in working example 1, which

indicates high toughness, and furthermore, compressive fracture strength is high as well. The above-mentioned properties appear to be based on zirconia, and when bentonite is added, a significant increase in plasticity and flexibility of the kneaded material is observed, and as a result, cracks inside the abrasive grains is reduced, and the ratio of the abrasive grains without cracks is increased.

[0016]

[Table I]

Working example and comparative examples	Amount added (wt%)			
	ZrO_2	Bentonite	MgO	PVA
Working example 1	3	0.3	0.2	0.4
Comparative example 1	3	—	0.2	0.4
Comparative example 2	—	0.3	0.2	0.4

[0017]

[Table II]

Working example and comparative examples	Amount included in abrasive grains (wt%)			Properties of abrasive grains					
	ZrO_2	SiO_2	MgO	Apparent specific gravity	Crystal size (μm)	Friability	Crack ratio (%)	Compressive fracture strength (kg/cm^2)	Vicker's hardness Hv 500 (kg/mm^2)
Working example 1	2.9	0.22	0.19	3.89	2.9	9.5	16.0	1624	1956
Comparative example 1	2.9	0.02	0.19	3.89	2.2	14.7	31.6	1508	1923
Comparative example 2	0.00	0.232	0.19	3.83	2.1	16.8	18.2	1368	1897

[0018] Working examples 2-5

The amount of zirconia added was changed as shown in Table III, and production of #12 abrasive grains was performed as in the case of working example 1. Properties of the abrasive grains produced are shown in Table III.

[0019] Comparative examples 3 to 7

As shown in Table III, the amount of zirconia added was changed to 3 wt% or less or 15 wt% or more. And production of #12 abrasive grains was performed as in the case of the above-mentioned working example 1. Properties of the abrasive grains produced are shown in Table III.

[0020]

[Table III]

Working examples and comparative examples	Amount of ZrO_2 added (wt%)	Amount included in abrasive grains (wt%)			Properties of abrasive grains	
		ZrO_2	SiO_2	MgO	Friability	Vicker's hardness Hv 500 (kg/mm^2)
Comp. ex. 2	0	0.0	0.23	0.19	16.6	1897
Comp. ex. 3	0.2	0.2	0.23	0.19	16.8	1907
Comp. ex. 4	1	1.0	0.23	0.19	13.5	1920
Work. ex. 1	3	2.9	0.22	0.19	9.5	1958
Work. ex. 2	5	4.7	0.21	0.18	8.6	1944
Work. ex. 3	8	7.4	0.21	0.18	8.3	1861
Work. ex. 4	10	9.0	0.21	0.18	7.4	1880
Work. ex. 5	15	13.0	0.20	0.17	9.6	1830
Comp. ex. 5	20	16.6	0.19	0.16	16.8	1614
Comp. ex. 6	25	19.9	0.18	0.15	16.5	1515
Comp. ex. 7	30	23.0	0.18	0.15	22.8	1401

[0021] Working examples 6 to 8 and comparative example 8

For the crushed and classified sintered alumina powder used as the raw material in working example 1, samples where 0.3 wt% of each clay of bentonite (product of Kanto Bentonite and Minerals Co., (Ltd.), Tenryu [from Higashi Kanbara region of Niigata Prefecture), kibushi clay (product of Sanage Ceramics Co., (Ltd.), super-fine powder [from Sanage of Nishikamo region of Aichi Prefecture), and frog eye clay (product of Matsubara Ceramics Co., (Ltd.), hydraulic elutriation product (from Seto region of Aichi Prefecture) was added, and a sample without a clay was used and 0.2 wt% of MgO (material used in working example 1), 0.4 wt% of PVA (material used in working example 1) and 26 wt% of water were added to each sample, and kneading was performed by a mixed muller. The load of the kneader was 7 to 8 A (AC) in all cases.

[0022] The degree of needle penetration by a clay hardness tester of Japan Glass Co., Ltd. that indicates the plasticity of the kneaded material was 13 in all cases where a clay was included, and 14 without a clay. Extrusion was performed for the kneaded materials by Tensilon (product of Toyo Baldwin Co., Ltd., UTM-10T-PL) at a crosshead speed of 5 mm/min to form a bar with a diameter of 3 mm. The start load and the maximum load of each case are shown in Table IV below.

[0023]

[Table IV]

Working examples and comparative examples	Clay used	Start load (kg)	Maximum load (kg)
Working example 6	Bentonite	400	960
Working example 7	Kibushi clay	430	1030
Working example 8	Frog eye clay	750	1180
Comparative example 8	None	850	1650

[0024] As shown in Table IV, excellent extrusion property was achieved in the order of bentonite

> kibushi clay > frog eye clay > none.

Comparative example 9

A crushed and classified sintered bauxite (d50=2 μm) was used as a raw material and 0.3 wt% PVA was added for the sintered bouxite and sintering was performed at a temperature in the range of 1400 to 1450°C for 1 hour and production of a sintered abrasive grains was performed as in the case of working example 1. The friability of the sintered abrasive grains was 10.3, and the Vicker's hardness was 1261 kg/mm^2, and the hardness is inferior to those of present invention.

[0025] As shown in Table III, a sample with a zirconia content in the range of 3 to 15 wt% exhibits a friability value of 10 or below, and high toughness. Furthermore, the Vicker's hardness of at least 1800 kg/mm^2 is retained as well.

[0026]

[Effect of the invention] The abrasive grains of the present invention includes a specific amount of alumina, zirconia, clay, and either magnesium oxide, cobalt oxide or nickel oxide. In this case, toughness can be significantly increased while high hardness is being retained (friability value can be significantly reduced) and cracks inside the abrasive grains can be reduced; thus, an alumina quality abrasive grains excellent properties as can be produced.

(19)【発行国】日本国特許庁（ＪＰ）

(19) [Publication Office] Japanese Patent Office (JP)

(12)【公報種別】公開特許公報（Ａ）

(12) [Kind of Document] Japan Unexamined Patent Publication (A)

(11)【公開番号】特開平７－２８６１６６

(11) [Publication Number of Unexamined Application] Japan Unexamined Patent Publication Hei 7 - 286166

(43)【公開日】平成７年（１９９５）１０月３１日

(43) [Publication Date of Unexamined Application] 1995 (1995) October 31 day

(54)【発明の名称】アルミナージルコニア質焼結砥粒およびその製造方法

(54) [Title of Invention] ALUMINA - ZIRCONIA QUALITY SINTERED ABRASIVE GRAIN AND ITS MANUFACTURING METHOD

(51)【国際特許分類第６版】

(51) [International Patent Classification 6th Edition]

C09K 3/14 550 D

H

B24D 3/00 320 A

C04B 35/10

【ＦＩ】

[FI]

C04B 35/10 E

【審査請求】未請求

[Request for Examination] Examination not requested

【請求項の数】３

[Number of Claims] 3

【出願形態】ＯＬ

[Form of Application] OL

【全頁数】５

[Number of Pages in Document] 5

(21)【出願番号】特願平６－８０６３８

(21) [Application Number] Japan Patent Application Hei 6 - 80638

(22)【出願日】平成６年（１９９４）４月１９日

(22) [Application Date] 1994 (1994) April 19 day

(71)【出願人】

(71) [Applicant]

【識別番号】０００００２００４

[Applicant Code] 000002004

【氏名又は名称】昭和電工株式会社

[Name] SHOWA DENKO K.K. (DB 69-057-0080)

【住所又は居所】東京都港区芝大門１丁目１３番９号

[Address] Tokyo Minato-ku Shiba Daimon 1-13-9

(72)【発明者】

(72) [Inventor]

【氏名】森元　久志

[Name] Forest Motohisa aspiration

【住所又は居所】長野県塩尻市大字宗賀１　昭和電工株

[Address] Inside of Nagano Prefecture Shiojiri City Oaza Soga 1

式会社塩尻工場内

Showa Denko K.K. (DB 69-057-0080) Shiojiri Works

(72)【発明者】

(72) [Inventor]

【氏名】金子　幸照

[Name] Kaneko　Yukiteru

【住所又は居所】長野県塩尻市大字宗賀１　昭和電工株式会社塩尻工場内大分研究所内

[Address] Inside of Oita Research Laboratory inside Nagano Prefecture Shiojiri City Oaza Soga 1　Showa Denko K.K. (DB 69-057-0080) Shiojiri Works

(74)【代理人】

(74) [Attorney(s) Representing All Applicants]

【弁理士】

[Patent Attorney]

(57)【要約】

(57) [Abstract]

【目的】　従来砥粒に比べて硬度を維持し、靭性を高め、耐摩耗性を向上し、高研削比のアルミナ－ジルコニア質焼結砥粒を提供する。

[Objective]　Until recently, it maintains hardness in compariso n with grit,raises toughness, abrasion resistance improves, offers alumina - zirconiaquality sintered abrasive grain of high grinding ratio.

【構成】　SiO_2 を多少含有し、靭性値を示すフライアビリティーの値が１５以下で、マイクロビッカース硬度が１８００kg／mm² 以上のアルミナ－ジルコニア質焼結砥粒。

[Constitution]　Value of friability which contains SiO2　more o r less, shows toughnessvalue being 1 5 or less, microvickers hardness alumina - zirconia quality sintered abrasive grain ofthe 1800 kg/mm2 or greater.

【特許請求の範囲】

[Claim(s)]

【請求項１】　実質的にアルミナ－ジルコニア質組成であって、

[Claim 1]　Being a alumina - zirconia quality composition subs tantially,

（１）ZrO_2 として３～１５wt%、

(1) As ZrO2　3 to 15 wt%,

（２）SiO_2 として０．０５～３．０wt%、

(2) As SiO2　0.05 to 3.0 wt%,

（３）ＭｇＯ，ＣｏＯおよびＮｉＯのうち少なくとも１種をこれらの表示換算

(3) Inside at least 1 kind of MgO, CoO and NiO these indication conversions

として０．１～３．０wt%含有し、靭性値を示すフライアビリティーの値が１５以下、荷重５００ｇでのマイクロビッカース硬度（Ｈｖ５００）が１８００kg／mm² 以上からなることを特徴とするアルミナ－ジルコニア質焼結砥粒。

Value of friability which 0.1 to 3.0 wt% it contains as, shows t oughness valuethe alumina - zirconia quality sintered abrasive grain which designates that microvickers hardness (Hv 500) withthe 1 5 or less and load 500g consists of 1800 kg/mm2 or greater as feature.

【請求項２】　純度９８wt%以上のアルミナ微粉末に対し、ジルコニアを３～１５wt%および粘土を焼結砥粒中にSiO_2 として０．０５～３．０wt%を加え、更に酸化マグネシウム、酸化コバルトおよび酸化ニッケルのうち少なくとも１種を０．１～３wt%添加し、混合後成型し、１５５０～１７５０℃にて焼成することを特徴とするアルミナ－ジルコニア質焼結砥粒の製造方法。

[Claim 2]　Vis-a-vis alumina fine powder of purity 98 wt% or gr eater, zirconia manufacturing method of alumina -zirconia quality sintered abrasive grain which designates that furthermore 0.1 to 3 wt% itadds inside at least 1 kind of magnesium oxide, cobalt oxide and nickel oxide in thesintered abrasive grain 3 to 15 wt% and clay including 0.05 to 3.0 wt% as SiO2, aftermixing molding does, calcines with 1550 to 1750 °C as feature.

【請求項３】　粘土がベントナイト、木節粘土、蛙目粘土、▲せっ▼器粘土、セリサイト粘土のうち少なくとも１種であることを特徴とする請求項２記載のアルミナ－ジルコニア質焼結砥粒の製造方法。

[Claim3] Clay bentonite (DANA 71.3.1a.1-2), Kibushi clay and frog eye clay, せ っ vessel clay, manufacturing method of alumina - zirconia quality sintered abrasive grain which isstated in Claim 2 which designates that it is inside at least 1 kind ofthe sericite clay as feature.

【発明の詳細な説明】

[Description of the Invention]

【０００１】

[0001]

【産業上の利用分野】本発明は重研削に適した高靭性の特性を有するアルミナ－ジルコニア質焼結砥粒に関する。

[Field of Industrial Application] This invention regards alumina - zirconia quality sintered abrasive grain which possesses thecharacteristic of high toughness which is suited for heavy grinding.

【０００２】

[0002]

【従来の技術】従来重研削用の砥粉はボーキサイトのようなアルミナ質物質の微粉を焼結した砥粒（特公昭３９－４３９８号、特公昭３９－２７６１２号、特公昭３９－２７６１３号、特公昭３９－２７６１４号）や溶融アルミナ－ジルコニア砥粒（特公昭３９－１６５９２号）やSiO_2，TiO_2，MgO，CaO等を含む溶融アルミナ－ジルコニア砥粒(特公昭５０－１３９８９号)や高純度アルミナ微粉に酸化マグネシウム等の結晶成長抑制剤を添加した砥粒（特開昭５２－１４９９３号）や高純度アルミナ微粉に酸化マグネシウム等の結晶成長抑制剤と、砥粒内クラックを低減させるために粘土を砥粒中に二酸化ケイ素として添加した砥粒（特開平４－２０５８６号）が知られている。また、特開昭５６－３２３６９号のようにゾルゲル法によるジルコニア、ハフニア、ニッケル、コバルト、亜鉛、マグネシア等を含むアルミナ質焼結砥粒も知られている。

[Prior Art] Until recently as for whetstone powder for heavy gri nding grit which sinters fine powder of alumina quality substance like bauxite (Japan Examined Patent Publication Sho 39 - 4398 number, Japan Examined Patent Publication Sho 39 - 27612 number, Japan Examined Patent Publication Sho 39 - 27613 number and Japan Examined Patent Publication Sho 39 - 27614 number)and fused alumina - zirconia grit (Japan Examined Patent Publication Sho 39 - 16592 number) and fused alumina - zirconia grit which includes theSiO_2, TiO_2, MgO, CaO etc (Japan Examined Patent Publication Sho 50 - 13989 number) and grit (Japan Unexamined Patent Publication Hei 4 - 20586 number) which it adds in grit as silicon dioxidehas been known grit which adds magnesium oxide or other crystal growth suppression agent to high purity alumina fine powder (Japan Unexamined Patent Publication Showa 52 - 14993 number) and theclay in order to decrease crack inside magnesium oxide or other crystal growth suppression agent and grit inthe high purity alumina fine powder. In addition, like Japan Unexamined Patent Publication Showa 56 - 32369 number also alumina quality sintered abrasive grain whichincludes zirconia, hafnia, nickel, cobalt, zinc and the magnesia etc by sol-gel method is informed.

【０００３】

[0003]

【発明が解決しようとする課題】ボーキサイト系焼結砥粒は、使用するボーキサイト(仮焼)中に、アルミナ（Al_2O_3）約８５%の他に、TiO_2，Fe_2O_3，SiO_2等の不純物を含んでいるため摩耗率が高いが、硬度が低い。溶融アルミナ－ジルコニア質砥粒は、原料を溶融した後、急冷固化、破砕、整粒して製品とするが、所定の優れた特性を有し必要粒度範囲の粒群を収率良く生産するのが困難である。また、重研削として用いられる高純度アルミナ質焼結砥粒は、硬度および機械的強度はまあまあ良いのに対して靭性が低く、時間当り研削量は大きいが、耐摩耗性が低いために、用途拡大の障害と

[Problems to be Solved by the Invention] As for bauxite sintere d abrasive grain, because in bauxite (calcining) which is used, TiO_2, Fe_2O_3, SiO_2 or other impurityis included in other than alumina (Al_2O_3) approximately 85 %, wear rate ishigh, but hardness is low. fused alumina - zirconia quality grit after melting starting material, quench solidification ,the fragmenting and granulating doing, makes product, but specified itpossesses characteristic which is superior and yield it is difficultwell to produce grain group of necessary grain size range. In addition, as for high purity alumina quality sintered abrasive grain which is used as the heavy grinding, as for

なっている。本発明の目的は、これらの課題を解決するために、硬度を最低限維持し、更に砥粒の靭性を高めることにより耐摩耗性を向上させ、それにより研削比向上を図り、また必要粒度範囲のものを押出し等により成形することにより、不要粒度を発生させないことを目的とするものである。

hardness and mechanical strength toughness is low vis-a-vis beinggood so-so, per hour amount of grinding is large, but because abrasion resistance is low, it hasbecome disorder of application enlargement. It is something which designates that in order to solve these problem,the hardness minimum limit it maintains objective of this invention, abrasion resistanceimproving furthermore by raising toughness of grit, it assuresgrinding ratio improvement with that, it does not generate unnecessarygranularity by forming in addition those of necessary grain size range with theextrusion etc, as objective.

【０００４】

【課題を解決するための手段】発明者は、上記の目的を達成すべく種々検討した結果、本発明を見出した。即ち、実質的にアルミナ－ジルコニア質組成であって、（１）ZrO_2として３～１５wt%、（２）SiO_2として０．０５～３．０wt%、（３）ＭｇＯ，ＣｏＯおよびＮｉＯのうち少なくとも１種をこれらの表示換算として０．１～３．０wt%含有し、靭性値を示すフライアビリティーの値が１５以下、荷重５００ｇでのマイクロビッカース硬度（Ｈｖ５００）が１８００kg／mm^2 以上からなることを特徴とするアルミナ－ジルコニア質焼結砥粒ならびにその製造方法として、純度９８wt%以上のアルミナ微粉末に対し、ジルコニアを３～１５wt%および粘土を焼結砥粒中にSiO_2として０．０５～３．０wt%を加え、更に酸化マグネシウム、酸化コバルトおよび酸化ニッケルのうち少なくとも１種を０．１～３wt%添加し、混合後成型し、１５５０～１７５０℃にて焼成することを特徴とするアルミナ－ジルコニア質焼結砥粒の製造方法を見出した。更に上記の該粘土がベントナイト、木節粘土、蛙目粘土、▲せっ▼器粘土、セリサイト粘土のうち少なくとも１種であることを特徴とするアルミナ－ジルコニア質焼結砥粒の製造方法も見出した。

[0004]

[Means to Solve the Problems] As for inventor, in order that ab ove-mentioned objective is achieved,the various as for result which was examined, this invention wasdiscovered. Namely, Being a alumina - zirconia quality composition substantially, being, As (1) ZrO2 3 to 15 wt%, As (2) SiO2 0.05 to 3.0 wt%, 0.1 to 3.0 wt% it contains with (3) MgO, CoO and inside at least 1 kind of theNiO as these indication conversions, Value of friability which shows toughness value 1 5 or less, alumina - zirconia quality sintered abrasive grain and manufacturing method which designate thatthe microvickers hardness (Hv 500) with load 500g consists of 1800 kg/mm2 or greater as feature doing, Vis-a-vis alumina fine powder of purity 98 wt% or greater, zirconia furthermore 0.1 to 3 wt% itadded inside at least 1 kind of magnesium oxide, cobalt oxide and nickel oxide in thesintered abrasive grain 3 to 15 wt% and clay including 0.05 to 3.0 wt% as SiO2 , aftermixing molding did, it discovered manufacturing method of alumina - zirconiaquality sintered abrasive grain which designates that it calcines with 1550 to 1750 °C asfeature. Furthermore above-mentioned said clay bentonite (DANA 71.3.1a.1-2), Kibushi clay and thefrog eye clay, せっ vessel clay, discovered also manufacturing method ofthe alumina - zirconia quality sintered abrasive grain which designates that it is theinside at least 1 kind of sericite clay as feature.

【０００５】本発明に使用するアルミナ原料微粉末の純度は９８wt%以上、好ましくは９９wt%以上で、該粒度は平均径d_{50}として好ましくは１０μｍ以下、より好ましくは２μｍ以下の微粉末である。純度が９８wt%未満では、他に添加する成分の調整に困難が生じ易く、均一性が問題になることがあり、粒度が１０μｍを越えると緻密な焼結体が得られ難いとともに焼結体の結晶サイズが増大し機械的強度が低下し好ましくない。また、本発明に使用するジルコニアは純度として９８wt%以上、d_{50}粒度として好ましくは１０μｍ以下、より好ましくは１μｍ以下の微粉末が好ましい。ジルコニア分の添加量は３～１５wt%が適しており、３wt%未満では砥粒の靭性向上の効果が十分でなく、また１５wt%を越えると硬度が著しく低下してしまう。また、焼結時の異常粒の結

[0005] As for purity of alumina starting material fine powder w hich is used for this invention with the98 wt% or greater and preferably 99 wt% or greater, as for said granularity it is a fine powder of preferably 10 mor less andthe more preferably 2 mor less as average diameter d50. purity is easy to occur under 98 wt%, difficulty in adjustmentof component which is added to other things, when there are times whenthe uniformity becomes problem, granularity exceeds 10 m, dense sinter isdifficult to be acquired and also crystal size of sinter increases andthe mechanical strength decreases and is not desirable. In addition, as for zirconia which is used for this invention fine powder ofthe preferably 10 mor less and more preferably 1 mor less is desirable as 98 wt% or greater and d50 granularity as thepurity. As for addition quantity of zirconia

晶抑制剤として砥粒組織中に０．１～３．０wt%含有されるように酸化マグネシウム、酸化コバルトまたは酸化ニッケルの少なくとも１種を添加する。０．１wt%未満では抑制効果が少なく、３．０wt%を超えると砥粒の硬度を低下し、機械的強度も劣るため好ましくない。これらの異常粒の結晶抑制剤も純度として９８wt%以上、d_{50}粒度としても好ましくは１０μｍ以下、より好ましくは５μｍ以下の微粉末が好ましい。本発明では砥粒の形状を円柱状等に成形するためにＰＶＡ、メチルセルロース（ＭＣ）等の有機バインダー、好ましくはその水溶性のものを０．２～３．０wt%程度入れるのが好ましいが、これだけでは十分な成形体の保形性が保たれない場合が多いので、本発明では必ず粘土を添加し、混練物の可塑性、柔軟性を向上させ、成形体のミクロクラック等を低減させる。

amount 3 to 15 wt% is suitable, when under the3 wt% effect of toughness improvement of grit not to be a fully, inaddition it exceeds 15 wt%, hardness decreases considerably. In addition, as 0.1 to 3.0 wt% contained in grit structure as crystal inhibitor of thefault grain when sintering, magnesium oxide, cobalt oxide or nickel oxide theat least 1 kind is added. When under 0.1 wt% supression effect is little, exceeds 3.0 wt% hardnessof grit it decreases, because also mechanical strength is inferior, it isnot desirable. fine powder of preferably 10 m or less and more preferably 5 m or less is desirable as 98 wt% or greater and thed50 granularity crystal inhibitor of these fault grains as purity. With this invention shape of grit 0.2 to 3.0 wt% extent water soluble things suchas PVA, methylcellulose (MC) or other organic binder and preferably it is desirable in order to formin cylinder etc to insert, but because among just this when automorphicof sufficient molded article is not maintained is many, with this invention be sure to addthe clay, plasticity of kneaded substance, softening to improve, to decreasethe micro crack etc of molded article.

【０００６】粘土には多くの種類があるが、特に本発明に対し混練物の可塑性を著しく向上させるものとして、ベントナイト、木節粘土、蛙目粘土、▲せっ▼器粘土、セリサイト粘土のうち少なくとも１種を使用するのが望ましく、このうちでは後述の実施例の結果等からして可塑性および乾燥後の強度から判断するとベントナント＞セリサイト粘土＞木節粘土＞蛙目粘土＞▲せっ▼器粘土の順でベントナイトが最も優れている。▲せっ▼器粘土でも粘土無添加の場合に比べて押出成形性等において優れており、押出成形性から見ても上記の順でベントナイトが最も好ましい。粘土の添加量は、焼結砥粒中にSiO_2として、０．０５～３．０wt%含有される量である。０．０５wt%未満では混練物の可塑性、柔軟性を向上させる効果が十分でなく、また、３．０wt%を超えるとガラス質やムライト質を生成し、砥粒の摩耗率を大きくしてしまうとともに、焼結により結晶が成長しすぎて強度が低下する。

[0006] There are many types in clay, but, Especially vis-a-vis t his invention plasticity of kneaded substance those whichimprove considerably doing, When bentonite (DANA 71. 3.1a.1-2), Kibushi clay and frog eye clay, せ っ vesselclay, it is desirable, among these mustard such as result of latermentioned Working Example て judges from strength after plasticity and drying touse inside at least 1 kind of sericite clay, vent nun jp7 > sericite clay > Kibushi clay > frog eye clay >.solid triangl bentonite (DANA 71.3.1a.1-2) most is superior in order of っ vessel clay. せ In comparison with in case of clay no addition we are superior even in theっ vessel clay in extrusion moldability etc, bentonite (DANA 71.3.1a.1-2) is most desirable inabove-mentioned order considered as extrusion moldability. addition quantity of clay 0.05 to 3.0 wt% is quantity which is contained inthe sintered abrasive grain as SiO_2 . Under 0.05 wt% plasticity of kneaded substance, softening effect whichimproves not to be a fully, in addition, when it exceeds 3. 0 wt%, asthe glass and mullite-like are formed, wear rate of grit isenlarged, crystal growing too much with sintering, strengthdecreases.

【０００７】本発明は上記の原料、即ち、アルミナ微粉末、ジルコニア、粘土および酸化マグネシウム等の異常粒結晶抑制剤を上記の範囲にて配合し、混合する。この場合、上記のようにＰＶＡ等の有機結合剤の水溶液、非水溶液を加えるのが好ましく、十分に混練後、円柱状等にて押出し等で成形する。成形は円柱状に限定するものでなく、形成方法も押出法に限定するものではない。その後、好ましくは１００～１５０℃の範囲にて乾燥し、焼成する。焼成温度は、１５５０～１７５０℃が適しており、１５５０℃より低温では緻密で高密度な焼結体が得られず、１７５０℃より高温では結晶サイズが大きす

[0007] This invention above-mentioned starting material, nam ely, combines alumina fine powder, the zirconia, clay and magnesium oxide or other fault grain crystal inhibitor in above-mentioned range,mixes. In this case, as description above aqueous solution of PVA or other organic binder, it isdesirable, in fully after kneading, forms with extrusion etc withsuch as cylinder to add nonaqueous solution. Formation not to be something which is limited in cylinder, is notsomething which also formation method limits in extrusion. after that, it dries in range of preferably 100 to 150 °C, calcines. As for sintering temperature, 1550 to 1750 °C is suitable, at

ぎて、靭性および機械的強度が低下する。砥粒とするためには焼成後、粉砕し、整粒するか、または乾燥後、解砕し、整粒し、所定の粒度品について焼成しても良い。

temperature which islower than 1550 ℃ highly dense sinter is not acquired with dense, at thetemperature which is higher than 1750 ℃ crystal size being too large,the toughness and mechanical strength decrease. In order to make grit, after calcining, powder fragment it does, the granulating does, or after drying, fracture does, granulating does, it isgood calcining concerning specified granularity item

【０００８】次に上記の製造方法により造られる本発明のアルミナ－ジルコニア質焼結砥粒について記す。本発明の砥粒は、上記配合原料割合の組成、即ち、実質的にアルミナ－ジルコニア質組成であって、ZrO_2として３～１５wt%、SiO_2として０．０５～３．０wt%、更にＭｇＯ，ＣｏＯおよびＮｉＯのうち少なくとも１種をこれらの表示換算で０．１～３．０wt%を含有した砥粒である。このような組成の砥粒は従来にもあったが、特徴ある上記の本発明の製造方法によって従来にない高靭性でかつ高い硬度の砥粒が初めて造れた。即ち、靭性値を示すフライアビリティーの値は１５以下であって、荷重５００ｇでのマイクロビッカース硬度（Ｈｖ５００）が１８００kg／mm² 以上からなることを特徴とするアルミナ－ジルコニア質焼結砥粒が得られた。

[0008] Next you inscribe concerning alumina - zirconia qualit y sintered abrasive grain of thethis invention which is made by above-mentioned manufacturing method. grit of this invention, composition of above-mentioned blended raw material ratio,namely, being a alumina - zirconia quality composition substantially, 0.05 to 3.0 wt% ,furthermore inside at least 1 kind of MgO, CoO and NiO is gritwhich contains 0.1 to 3.0 wt% with these indication conversions as the3 to 15 wt% and SiO2 as ZrO2 . As for grit of this kind of composition it was even former and it couldmake grit of high hardness for first time with high toughnesswhich is not former, but with manufacturing method of above-mentioned this inventionwhich is feature. Namely, being a 1 5 or less, alumina - zirconia quality sintered abrasive grain whichdesignates that microvickers hardness (Hv 500) with load 500g consists of 1800 kg/mm2 or greater asfeature acquired value of friability which shows toughness value.

【０００９】なお、上記のフライアビリティーの測定法は以下の通りである。該測定法はＪＩＳ　Ｒ６１２８（人造研削材のじん性の試験方法）に準拠する方法であるが、多少以下のように条件を変えている。

[0009] Furthermore, measurement method of above-mentioned friability is as follows. said measurement method is method which conforms to JIS R6128 (test method of toughness of artificial abrasive), but like thesome or less condition is changed.

（１）試料である被測定の砥粒を約３００ｇを量り採り、各粒度の該ＪＩＳに規定された標準網ふるいを用い、ロータップ試験機にて５分間ふるい分けする。３段目ふるいに留まった試料の全量を更に５分間ふるい分け、更に３段目ふるいに留まった試料を試験試料とする。

(1) Grit of suffering measurement which is a sample you measur eapproximately 300g and take, 5 min screening you do with Rotap tester makinguse of standard network sieve which is stipulated in said JIS of each granularity. total amount of sample which remains in third step sieve furthermore the5 min screening, furthermore sample which remains in third step sieve is designatedas test sample.

（２）試験試料１００ｇを０．１ｇまで正確に量り、１１４mmφの１リットルのボールミルに入れ、更に３／４インチのスチールボール２kgをボールミルに入れる。

(2) You measure test sample 100g accurately to 0.1 g, insert in ball mill ofthe 1 liter of 114 mm diameter, furthermore insert steel ball 2 kg of 3/4 inch inthe ball mill.

（３）ミル回転数９５rpm で１５分間試験試料を粉砕する。その後、ボールミル内容物を取り出し、粉砕試料を回収する。

(3) 15 min test sample powder fragment is done with mill rotati on rate 95 rpm . after that, it removes ball mill contents, powder fragment sample recovers.

【００１０】（４）粉砕試料を上記と同じ標準網ふるいを用いロータップ試験機によって５分間ふるい分ける。

[0010] (4) Powder fragment sample 5 min is screened due to R otap tester making use of thesame standard network sieve as description above.

（５）各段のふるいに留まった試料を正確に量る。１～

(5) Sample which remains in sieve of each step is measuredaccur

４段目ふるいに留まった試料量の合計を試験試料量１００ｇから差し引いて、その値をフライアビリティー値とする。即ち、４段目ふるい下がフライアビリティー値となり、その値が小さい程、靭性が高いことになる。

【００１１】

【実施例】以下に実施例および比較例にて本発明を詳説する。

実施例１

仮焼アルミナ（純度９９．５wt%、比表面積５m^2／g）の粉砕分級粉（d_{50}＝１．９９μm、d_{10}＝０．４４μm、d_{90}＝７．７６μm）に対し、表１に示すようにジルコニア（昭和電工製ＲＺ－Ｎ、＃６０００Ｆ；d_{50}＝０．８９μm）を３wt%、ベントナイト（関東ベントナイト鉱業（株）製、天竜）を０．３wt%添加、更に酸化マグネシウム（協和化学工業製キョーワマグ３０、純度９９wt%、d_{50}＝４．５１μm）０．２wt%配合し、バンイダーとしてＰＶＡを０．４wt%加えるとともにその総重量に対し２６wt%の水に入れ、ミックスマーラーにて混練し、真空式押出機を用いてＪＩＳ　Ｒ６００１－１９８７の規定の粒度＃１２の砥粒にするため２．２mmφの孔径を有する口金より押出し、１５０℃で１時間乾燥した。この乾燥品を２～６mmの長さに切断し、ロータリーキルンにて１７００℃で１時間焼成した。

【００１２】かくして得られた砥粒の特性を表２に示す。粒内クラックは円柱状の中心を長さ方向にスライスした断面部を１０倍の顕微鏡で観察し、クラックが一つでもあった粒の割合を含有率として表示した。また、圧壊強度は〔破壊荷重〕／〔砥粒の投影面積〕で求めた値であり、結晶サイズはＳＥＭより測定した。

【００１３】比較例１

表１に示すようにベントナイトを添加せず、その他の条件等を実施例１と同一として粒度＃１２の砥粒を得た。その特性値等を表２に示す。砥粒中のSiO_2量はアルミナ原料の不純物から混入したものである。

【００１４】比較例２

表１に示すようにZrO_2を添加せず、その他の条件等を実施例１と同一として粒度＃１２の砥粒を得た。その

ately. Deducting total of sample weight which remains in 1 to fourth step sieve from thetest sample quantitative 100g, it designates value as friability value. Namely, when under of fourth step sieve reaches friability value, that value issmall, it means that toughness is high.

[0011]

[Working Example(s)] Below this invention detailed explanatio n is done with Working Example and Comparative Example .

Working Example 1

In milling and classification powder (d50=1.99 m, d10=0.44 m and d90=7.76 m) of calcining alumina (purity 99.5 wt% and specific surface area 5 m2/g) to confront, Way it shows in Table 1, zirconia (Showa Denko K.K. (DB 69-110-9268) make RZ - N, #6000F; d50=0.89 m) 3 wt%, bentonite (DANA 71.3.1a.1-2) (Kanto bentonite (DANA 71.3.1a.1-2) mining Ltd. make, Tenryu) is combined 0.3 wt% addition, furthermore magnesium oxide (Kyowa Chemical Industry Co. Ltd. (DB 69-068-6670) make Kyowamag 30, purity 99 wt% and d50=4.51 m)0.2 wt%, PVA 0.4 wt% is added as as binder - you inserted inthe water of 26 wt% vis-a-vis total weight, kneaded with mix muller, inorder to make grit of granularity #12 of rule of JIS R6001 - 1987, makinguse of vacuum type extruder from die which possesses pore diameter of 2.2 mm diameterthe 1 hour you dried with extrusion and 150 °C. It cut off this dry product in length of 2 to 6 mm, with rotary kiln the1 hour calcined with 1700 °C.

[0012] Characteristic of grit which it acquires in this way is sho wn in theTable 2. crack inside grain cylindrical center in longitudinal direction observed cross section partwhich slice is done with microscope of 10 times, indicated theratio of grain where crack is one as content. In addition, compressive fracture strength projected surface area) (of breaking load) / (grit with was valuewhich was sought, it measured crystal size from SEM.

[0013] Comparative Example 1

As shown in Table 1, bentonite was not added, grit of granularit y #12was acquired other condition etc as same as Working Example 1. characteristic value etc is shown in Table 2. amount of SiO2 in grit is something which is mixed from impurity ofthe alumina starting material.

[0014] Comparative Example 2

As shown in Table 1, ZrO2 was not added, grit of granularity # 12was acquired other condition etc as same as Working

特性値等を表２に示す。

Example 1. characteristic value etc is shown in Table 2.

【００１５】表２より分かるように実施例１のものはフライアビリティーが極めて小さく、靭性が高いことを示しており、かつ圧壊強度も高い。この両特性はジルコニアに負うところが大きいと思われるが、ベントナイトを添加する効果として混練物の可塑性、柔軟性が数段向上し、その結果として砥粒内のクラックが少なくなるとともにクラックのない砥粒の比率が高くなっている。

[0015] As Table 2 compared to understood, those of Working Example 1 friabilityquite are small, we have shown fact that toughness is high, at thesame time also compressive fracture strength is high. This both characteristics is thought that place where it owes to zirconia islarge, but plasticity of kneaded substance, softening several steps improves as theeffect which adds bentonite (DANA 71.3.1a.1-2), crack inside grit decreases, asas result ratio of grit which does not have crack hasbecome high.

【００１６】

[0016]

【表１】

[Table 1]

実施例No.	添加量 (wt%)			
比較例No.	ZrO_2	ベントナイト	MgO	PVA
実施例１	３	０．３	０．２	０．４
比較例１	３	―	０．２	０．４
比較例２	―	０．３	０．２	０．４

【００１７】

[0017]

【表２】

[Table 2]

実施例No. 比較例No.	砥粒中含有量 (wt%) ZrO_2	SiO_2	MgO	砥粒特性 見掛比重	結晶サイズ (μm)	フライアビリティー	クラック含有率 (%)	圧壊強度 (kg/cm²)	ビッカース硬度 Hv 500 (kg/mm²)
実施例１	2.9	0.22	0.19	3.89	2.9	8.5	16.0	1624	1956
比較例１	2.9	0.02	0.19	3.89	2.2	14.7	31.6	1508	1923
比較例２	0.00	0.23	0.19	3.83	2.1	18.6	18.2	1368	1897

【００１８】実施例２～５

[0018] Working Example 2 to 5

表３に示すようにジルコニア添加量を変えたこと以外は実施例１と同一条件で#１２の砥粒を造った。得られた砥粒の特性を表３に示す。

As shown in Table 3, other than thing which changed zirconia a ddition quantitythe grit of #12 was made with Working Example 1 and identical condition. characteristic of grit which

is acquired is shown in theTable 3.

【0019】比較例3～7

表3に示すようにジルコニア添加量を3wt%未満および15wt%を越えるものを検討した。その他の条件は実施例1と同一条件で#12の砥粒を造った。得られた砥粒の特性を表3に示す。

[0019] Comparative Example 3 to 7

As shown in Table 3, zirconia addition quantity under 3 wt% and those whichexceed 15 wt% were examined. Other condition made grit of #12 with Working Example 1 and theidentical condition. characteristic of grit which is acquired is shown in theTable 3.

【0020】

[0020]

【表3】

[Table 3]

実施例No 比較例No	ZrO_2 添加量 (wt%)	砥粒中含有量(wt%)			砥粒特性	
		ZrO_2	SiO_2	MgO	フライアビリティー	ビッカース硬度 (Hv500) (kg/mm²)
比較例2	0	0.0	0.23	0.19	18.6	1897
比較例3	0.2	0.2	0.23	0.19	16.8	1907
比較例4	1	1.0	0.23	0.19	18.5	1920
実施例1	3	2.9	0.22	0.19	9.5	1956
実施例2	5	4.7	0.21	0.18	8.6	1944
実施例3	8	7.4	0.21	0.18	8.3	1861
実施例4	10	9.0	0.21	0.18	7.4	1880
実施例5	15	13.0	0.20	0.17	9.6	1830
比較例5	20	16.6	0.19	0.16	16.8	1614
比較例6	25	19.9	0.18	0.15	16.5	1515
比較例7	30	23.0	0.18	0.15	22.6	1401

【0021】実施例6～8および比較例8

ベントナイト（関東ベントナイト鉱業（株）（天竜）〔新潟県東蒲原郡産〕、木節粘土（枝下窯業（株）超微粉品〔愛知県西加茂郡猿投町枝下産〕）および蛙目粘土（松原セラミック（株）水簸品〔愛知県瀬戸市産〕）3種の粘土と粘土を添加しない条件で、実施例1にて原料として使用した仮焼アルミナの粉砕分級粉に対し外割にてそれぞれの粘土を0．3wt%、MgO(実施例1と同じ品物)0．2wt%、PVA（実施例1と同一物）0．4wt%および水26wt%配合し、ミックスマーラーにて混練した。混練機の負荷は粘土無添加の場合も含め総て7～8A（交流）であった。

[0021] Working Example 6 to 8 and Comparative Example 8

Bentonite (DANA 71.3.1a.1-2) (Kanto bentonite (DANA 71. 3.1a.1-2) mining Ltd. (Tenryu) (Niigata Prefecture Higashi Kanbara-gun product), With clay of Kibushi clay (the refractory industry Ltd. ultrafine powder item (product under Aichi Prefecture Nishikamo-gun Sanagechobranch)) under branch and frog eye clay (the Matsubara ceramic Ltd. hydraulicelutriation item (Aichi Prefecture Seto city product)) 3 kinds and does not add theclay condition which, With Working Example 1 0.3 wt% and MgO (the same article as Working Example 1)0.2 wt%, PVA (Working Example 1 and same compound) 0.4 wt% and water 26 wt% itcombined respective clay at outside percentage as starting materialvis-a-vis milling and classification powder of calcining alumina which you use, kneaded with the mix muller . In case of clay no addition it included and load of kneader it was a7 to 8A(alternating current) entirely.

【0022】混練物の可塑性を示す日本碍子製クレーハードネステスターによる針入度は、粘土を添加したものは総て13で、無添加のものは14であった。混練物をテンシロン（TOYO　BALDWIN社製UTM－1

[0022] As for needle penetration due to NGK Insulators Ltd. (D B 69-055-9968) make clay hardness tester which shows plasticity ofthe kneaded substance, as for those which add clay with all 13, as forthose of no addition it was a 14. kneaded

０Ｔ－ＰＬ型）を使用し、３mmφの棒をクロスヘッドスピード５mm/minにて押出した。その際のそれぞれの出始め荷重と最大荷重を表４に示す。

substance you use Tensilon (TOYO BA LDWIN supplied UTM - 10T - P L shape), rod of 3 mm diameter extrusion are withthe crosshead speed 5 mm/min. At that case respective start load and maximum load are shown in theTable 4.

【００２３】

[0023]

【表４】

[Table 4]

実施例No. 比較例No.	使用粘土	出始め荷重 〔kg〕	最大荷重 〔kg〕
実施例６	ベントナイト	４００	９６０
実施例７	木節粘土	４３０	１０３０
実施例８	蛙目粘土	７５０	１１８０
比較例８	無使用	８５０	１６５０

【００２４】表４より分かるようにベントナイト＞木節粘土＞蛙目粘土＞無使用（添加）の順で押出性は優れていた。

[0024] As understood from Table 4, extrusion behavior was sup erior in order of thebentonite (DANA 71.3.1a.1-2) > Kibushi clay > frog eye clay > non use (Addition).

比較例９

Comparative Example 9

原料に仮焼ボーキサイトの粉砕分級品（d_{50}＝２μｍ）、バンダーは仮焼ボーキサイトに対してＰＶＡを０．３wt%、ロータリーキルンにて１４００～１４５０℃で１時間焼成すること以外は実施例１と同一条件で焼結砥粒を得た。得られた焼結砥粒のフライアビリティーの値は１０．３で、ビッカース硬度は１２６１kg／mm² であり、本願発明のものに比べて、特に硬度が低いことが分かる。

In starting material milling and classification itemof calcining b auxite (d50=2 m), Vanda coerulea Griff. ex Lindl. - PVAwith 0.3 wt% and rotary kiln with 1400 to 1450 °C 1 hour other than thething which calcines acquired sintered abrasive grain with Working Example 1 and identical conditionvis-a-vis calcining bauxite. as for value of friability of sintered abrasive grain which is acquiredwith 10.3, as for Vicker's hardness it is a 1261 kg/mm2 , it understands thatthe especially hardness is low in comparison with those of invention of this application.

【００２５】表３よりジルコニアの含有量が３～１５wt%の範囲のものが、フライアビリティーの値が１０以下とボーキサイト系焼結砥粒のそれより小さく、靭性が高いことを示している。更に、ビッカース硬度が１８００kg／mm² 以上を維持している。

[0025] Table 3 compared to content of zirconia those of range of the3 to 15 wt%, value of friability are smaller than that of 1 0 or below and thebauxite sintered abrasive grain, fact that toughness is high has been shown. Furthermore, Vicker's hardness has maintained 1800 kg/mm2 or greater.

【００２６】

[0026]

【発明の効果】本発明の砥粒はアルミナにジルコニアと粘土および酸化マグネシウム、酸化コバルトもしくは酸化ニッケルのうちいずれかを特定量配合することにより、高硬度を保ちながら靭性を大幅に向上させ（フライアビリティー値は大幅に減少）、更に砥粒内クラックを減少させることができるため、アルミナ質焼結砥粒として優れた特性をいろいろ有するものである。

[Effects of the Invention] Grit of this invention while maintaini ng high hardness, by certain amountcombining inside any of zirconia and clay and magnesium oxide ,the cobalt oxide or nickel oxide in alumina, toughness greatly improving,because (As for friability value greatly decrease), furthermore it can decrease crack inside grit, issomething which variety it possesses characteristic which is superioras alumina quality sintered abrasive grain.